

PATENT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE



Applicants: Yamanaka et al.

Examiner: Kruer, K.

Serial No.: 08/855,905

Group Art Unit: 1773

Filed: May 14, 1997

Docket: 443-17

For: SYNTHETIC PAPER MADE OF
STRETCHED POLYPROPYLENE FILM

Assistant Commissioner for Patents
Washington, D.C. 20231

DECLARATION

Sir:

I, Masaaki Yamanaka, do hereby declare:

1. I am one of the joint inventors of the present U.S. application;
2. I have been engaged in research and development of synthetic paper at Oji-Yuka Synthetic Paper Co., Ltd. since 1969;
3. I am an inventor of the following U.S. Patents: U.S. Patent Nos. 4,097,645; 4,340,639; 4,341,880; 4,418,112; 4,420,530; 4,472,227; 4,483,965; 4,663,216; 4,705,719; 4,795,676; 4,986,866; 5,254,302; 5,332,542; 5,409,754 and 5,670,225 and the corresponding foreign patents such as Japanese and European patents;
4. The experimentation outlined in the present U.S. application, the results thereof being documented in Tables 1 and 2 therein, was carried out with my involvement, direction and supervision;

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5. The following additional Experiments 1 and 2, the results presented in accompanying Tables 1-3, was also carried out with my involvement, direction and supervision:

Experiment 1

A duplicate experiment was made on Example 2 of U.S. Patent No. 4,318,950 (invention made by the parent company of Oji-Yuka Synthetic Paper Co., Ltd., which is one of the present assignees (This technique has been transferred to Oji-Yuka Synthetic Paper Co., Ltd.)).

A resin composition (A) comprising 90 parts by weight of NOVATEC PP MA-8 (polypropylene produced by Japan Polychem Corporation; melting point: 164°C), 10 parts by weight of NOVATEC HD HJ580 (high density polyethylene produced by Japan Polychem Corporation; melting point: 134°C), 5 parts by weight of clay having a particle diameter of 1 μ m produced by ENGELHARD MINERALS & CHEMICALS CO., 1.0 parts by weight of calcium stearate as a stabilizer, 0.1 parts by weight of TOPANOL (1,3,5-tris(2-methyl-4-hydroxy-5-t-butyl phenol)butane)), 0.3 parts by weight of NYMEEN S-210 (dispersant produced by NOF Corp.) and 0.3 parts by weight of PHOSPHANOL RL-210 (revised name of "PHOSPHANOL SM-1", produced by TOHO Chemical Inc.) as a low-molecular weight antistatic agent was melt-kneaded through an extruder, and then extruded through a die into a sheet at a temperature of 250°C. The sheet thus formed was cooled to a temperature of about 50°C.

Subsequently, the sheet was heated to a temperature of about 140°C where

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it was then longitudinally oriented by a factor of 2 utilizing the difference in circumferential speed between rolls to obtain a longitudinally-oriented film. Separately, a resin composition (B) comprising 80 parts by weight of NOVATEC PP MA-8 (polypropylene produced by Japan Polychem Corporation; melting point: 165°C), 20 parts by weight of NOVATEC HD HJ580 (high density polyethylene produced by Japan Polychem Corporation; melting point: 134°C), 5 parts by weight of clay having a particle diameter of 1 μ m produced by ENGELHARD MINERALS & CHEMICALS CO., 1.0 parts by weight of calcium stearate as a stabilizer, 0.1 parts by weight of TOPANOL (1,3,5-tris(2-methyl-4-hydroxy-5-*t*-butyl phenol)butane)), 0.3 parts by weight of NYMEEN S-210 (dispersant produced by NOF Corp.) and 1.0 parts by weight of PHOSPHANOL RL-210 (revised name of "PHOSPHANOL SM-1", produced by TOHO Chemical Inc.) as a low-molecular weight antistatic agent was separately melt-kneaded through two extruders, and then extruded through a die onto the respective surface of the foregoing longitudinally-oriented film to obtain a paper-like layer (B)/base layer (A)/paper-like layer (B).

The three-layer film (B/A/B) was introduced into a tenter oven where it was then heated to a temperature of 160°C. The film was then oriented crosswise by a factor of 5 at a temperature of 150°C using the tenter. Subsequently, the film was thermally set at a temperature of 140°C. The film was subjected to corona discharge treatment at 70 W/m²/min on the paper-like layer (B layer) side thereof, cooled to a temperature of 55°C, and then slit at the edge thereof to obtain a three-layer synthetic paper having a thickness of 170 μ m (B/A/B = 60/50/60 μ m). The synthetic paper thus

obtained was a composite film comprising a biaxially-oriented base layer and two uniaxially-oriented paper-like layers (surface layers).

Experiment 2

A uniaxially-oriented paper was prepared in the same manner as in Example 1, except that instead of PHOSPHANOL SM-1, PHOSPHANOL RL-210 (produced by TOHO Chemical Inc.) was used as an antistatic agent and the incorporation of the resin component in the surface layer was effected as set forth in Table 2. During the preparation of the synthetic paper, the resin became sticky to the molding roll at the molding step. Thus, the resin could be hardly molded.

Table 3 shows the results of the evaluation of surface resistivity and offset printability of the synthetic papers obtained by Experiments 1 and 2 according to the method described in the present application. Referring to surface resistivity, Experiment 1 (low-molecular weight antistatic agent content: 0.3 parts) shows some improvement ($6 \times 10^{13} \Omega$) before rinsing but shows deterioration ($6 \times 10^{15} \Omega$) after rinsing. Experiment 2 (low-molecular weight antistatic agent content: 16.7 parts) shows a surface resistivity as high as $4 \times 10^{11} \Omega$ before rinsing but shows a drastic deterioration ($5 \times 10^{15} \Omega$) after rinsing. This is presumably because the low-molecular weight antistatic agent elutes out of the surface of the film during rinsing. Referring to adhesion of ink, both Experiments 1 and 2 evaluated only fair and thus are practically unacceptable. Referring to feedability and dischargeability, both Experiments 1 and 2 evaluated poorly and are liable to frequent trouble in feed and discharge. Thus, Experiments 1 and 2 are practically unacceptable;

6. I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

1999. 10. 6
Date

Masaaki Yamanaka
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Table 1

| Components: Paper-like layer | | | | | | | | | |
|------------------------------|-------|--------|-------|------------|-------------|-------------------|-------|------------------|-------|
| Resin | Parts | Filler | Parts | Stabilizer | Parts | Anti-static agent | Parts | Dispersing agent | Parts |
| Ex.1 | PP*1 | 80 | clay | 5 | Ca-stearate | 1.0 | 0.3 | NYMEEN S-210 | 1.0 |
| | PE | 20 | | TOPANOL | 0.1 | | | | |
| Components: Base layer | | | | | | | | | |
| Resin | Parts | Filler | Parts | Stabilizer | Parts | Anti-static agent | Parts | Dispersing agent | Parts |
| Ex.1 | PP*2 | 90 | clay | 5 | Ca-stearate | 1.0 | 0.3 | NYMEEN S-210 | 0.3 |
| | PE | 10 | | TOPANOL | 0.1 | | | | |

PP*1: NOVATEC.PP, MA-3, polypropylene produced by Japan Polychem Corporation (melting point: 165°C)

PP*2: NOVATEC.PP, MA-8, polypropylene produced by Japan Polychem Corporation (melting point: 164°C)

PE: NOVATEC.HD, HJ580, high density polyethylene produced by Japan Polychem Corporation (melting point: 134°C)

clay: clay having a particle diameter of 1 μ m produced by ENGELHARD MINERALS & CHEMICALS CO. PHOSPHANOL SM-1: PHOSPHANOL RL-210 (revised name of PHOSPHANOL SM-1), produced by TOHO Chemical, was used.



NYTEEN S-210: produced by NOF Corp.

Table 2

| | Final composition of surface layer | | | Molding/stretching/surface treatment | | |
|-------|--|--------------------------|---------------------------------------|--------------------------------------|------------------------------------|--------------------------------|
| | Resins (100 parts) | | Fine inorganic particles (E) | Thickness (μm) | Stretching of surface layer | of |
| | PP | Modif- ied PP (D1) | CaCO ₃ TiO ₂ | front/core/ back | Uni- biaxial stret- ching | or Stret- ching ratio |
| Ex. 1 | Blended amount is set forth in Table 1 | | | 60/50/60 | uniaxial | 5 |
| Ex. 2 | 72.3 | 16.7 | 5.5 | 72.7 | 9.1 | 20/60/20 |
| | | | | | uniaxial | 8 |
| | | | | | uniaxial | corona |



Table 3

| | Evaluation | | |
|-------|----------------------|----------------------|---------------------|
| | Surface resistivity | | Offset printability |
| | (a) | (b) | Ink adhesion |
| Ex. 1 | 6 x 10 ¹¹ | 6 x 10 ¹³ | Fair |
| Ex. 2 | 4 x 10 ¹¹ | 5 x 10 ¹³ | Fair |
| | | | Poor |
| | | | Poor |

| Suitability for paper feed/discharge | |
|--------------------------------------|--|
| Poor | |
| Poor | |